SYNTHESIS OF NANOCRYSTALLINE BISMUTH TITANATE PHOTOCATALYSTS VIA MODIFIED HOT INJECTION METHOD

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For Mom, Dad, Sis, and/f/

Thank you for your everlasting love and support

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ABSTRACT

Nanocrystalline bismuth titanate materials were successfully synthesized via modified hot injection method. The modified method used aqueous solution of nitric acid instead of non coordinating solvent as the reaction solvent which allowed a lower reaction temperature at 130^{o}C. XRD and FESEM analyses showed that the synthesized material crystallized in a cubic structure with *Fm3m* space group with average particle size of 7.9 nm. The effect of heating temperature showed that bismuth titanate with two space groups of *Fm3m* and *I23* were obtained after heating at 400°C for 3 hours. Interestingly, the mixed phase bismuth titanate materials have the lowest band gap energy of 2.57 eV and they showed the highest photocatalytic activity in phenol degradation UV light for 12 hours. The effect of ageing time on physico-chemical properties showed particle size of the materials increased with increasing of ageing time. As a result, bismuth titanate with 2 hours of ageing time was the best photocatalyst due to its small particle size of 6.4 nm. Similarly, surfactant content used did not affect phase formation of the materials but affected the particle size. The highest surface area of 20.2 m^2/g was observed in the bismuth titanate material synthesized using oleic acid to bismuth mole ratio of 1.46:1 and it had contributed to its high photocatalytic activity of 87% phenol degradation. In order to further examine the photocatalytic activity of the nanocrystalline bismuth titanate, bismuth titanate of different bismuth to titanium mole ratios (10:1 to 18:1) were synthesized. XRD results strongly suggested the formation of solid solution as all the materials crystallized in cubic structure with *Fm3m* space group. Bismuth titanate with bismuth to titanium mole ratio of 10:1 has achieved the highest phenol degradation percentage of 88% due to smaller particle size as well as higher mole ratio of titanium content in the material.

ABSTRAK

Bismut titanat berhablur nano telah berjaya disintesis melalui kaedah sintesis suntikan panas yang diubahsuai. Kaedah terubah suai menggunakan larutan akueus asid nitrik untuk menggantikan pelarut bukan koordinasi sebagai pelarut tindak balas bagi mengurangkan suhu tindak balas kepada 130°C. Analisis XRD dan FESEM menunjukkan bahan yang disintesis berhablur dalam sistem kiub dengan kumpulan ruangan *Fm3m* dengan purata saiz zarah sebanyak 7.9 nm. Kesan pemanasan menunjukkan bismut titanat wujud dalam dua fasa kumpulan ruangan iaitu *Fm3m* dan *I23* selepas dipanaskan pada 400°C selama 3 jam. Menariknya, fasa campuran bismut titanat tersebut mempunyai leluang jalur tenaga yang rendah iaitu 2.57 eV, lalu menunjukkan kadar fotodegradasi fenol yang tinggi di bawah sinaran UV selama 12 jam. Kajian kesan masa penuaan terhadap ciri-ciri fizikal-kimia menunjukkan saiz zarah bahan meningkat dengan peningkatan masa penuaan. Oleh yang demikian, bismut titanat dengan 2 jam masa penuaan sahaja adalah fotomangkin terbaik disebabkan saiz zarah yang kecil iaitu 22 nm. Seperti kesan masa penuaan, kuantiti surfaktan tidak mempengaruhi pembentukan fasa tetapi memberi kesan terhadap saiz zarah. Luas permukaan tertinggi sebanyak 20.2 m^2/g yang diperolehi pada bismut titanat yang disintesis menggunakan nisbah mol asid oleik kepada bismut 1.46:1, seterusnya menyumbang kepada aktiviti fotomangkin yang tinggi iaitu 87% dalam fotodegradasi fenol. Demi meningkatkan lagi aktiviti fotomangkin, bismut titanat yang berbeza nisbah mol bismut kepada titanat (10:1 hingga 18:1) telah disintesis. Corak XRD menunjukkan pembentukan pepejal larutan dalam semua bahan yang disintesis berhablur dalam sistem kuib dengan kumpulan ruangan *Fm3m*. Bismut titanat dengan nisbah mol bismut kepada titanat 10:1 mencapai fotodegradasi fenol yang tertinggi iaitu 88% disebabkan oleh saiz zarah yang kecil dan juga kandungan nisbah mol titanat yang tinggi di dalam bahan tersebut.